The University of Chicago

A STUDY OF THE METALLIC STATE AND THE MAGNETIC SUSCEPTIBILITIES OF SODIUM IN LIQUID AMMONIA

A DISSERTATION SUBMITTED TO THE FACULTY OF THE DIVISION OF THE PHYSICAL SCIENCES IN CANDIDACY FOR THE DEGREE OF DOCTOR OF PHILOSOPHY

DEPARTMENT OF CHEMISTRY
1934

By
HENRY GEORGE THODE

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The writer wishes to acknowledge his indebtedness and express sincere thanks to Dr. Simon Freed for his kindly suggestions and help in various ways. He also wishes to thank Dr. W. C. Johnson for various suggestions and use of apparatus.

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INTRODUCTION

The present theories of the metallic state are based on the assumption that the metal consists of a free electron gas, or in the more highly refined theories, an electron gas bound to centers of minimum potential energy in the regions of the positive ions. The electron gas constitutes the conducting electrons in the metal, or all those electrons not in closed shells. The theories further assume that the electrons follow the Pauli (1) - Fermi (2) Statistics with regard to their distribution of energy, momentum, and spin.

With the spin of the electron is associated its intrinsic magnetism. There are two orientations of this spin. In one the magnetism of two electrons cancel, and in the other they reinforce. By studying the magnetism and consequently the spin, there is thrown open the possibility of examining the whole statistics experimentally. Actual metals do not permit this because temperatures of 10000°A are needed in order to follow the magnetism of the metal to the critical point where the theories indicate a change in the spin orientation. The Fermi theory involves temperature and molal volume as significant parameters. In metals the differences in molal volume are too slight to make much difference in the distribution of energy or spin among the electrons, and consequently any change in distribution with temperature would become marked only above the boiling points of the metals. By dissolving sodium in liquid ammonia, it was possible to study the metallic state by varying at will the average distance between the atoms, or the electron density.

The magnetic susceptibility seemed to be especially qualified to indicate those interactions between electron spins to which the metallic state is ascribed, since the spin has a magnetic moment associated with it.

THEORETICAL DISCUSSION

In the new statistics, momentum space is divided into small cells, of which the centers give the values of the momenta corresponding to the different energy states. The Fermi statistics postulates a limitation on the number of electrons which can be in the same cell. This postulate is based on the Pauli-exclusion principle, which states that no two electrons may have the same four quantum numbers.

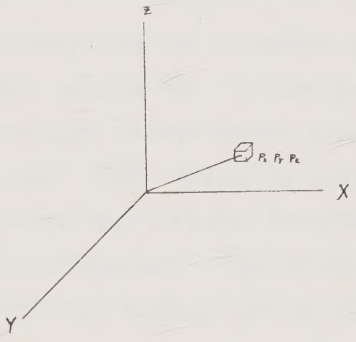

Fig. 1

It follows that two electrons in the same cell must have opposite spins and thus neutralize each other magnetically.

On this basis Fermi (2) and Dirac (3) independently worked out a distribution function for free electrons:

$$f = \frac{b}{h^3} \frac{1}{e^{(E - W_c)/KT} + 1}$$

where E is the energy of the electrons, W_c is their critical energy and h is Planks constant.

At the absolute zero the exponential term is either infinite or zero according as E is greater or less than W_c. When E is greater than W_c, the distribution function approaches the form

of the classical statistics. It turns out that up to the critical energy

$$W_c = \frac{h^2}{2m} \left(\frac{3n}{8\pi V}\right)^{2/3}$$

where $\frac{n}{V}$ is the number of electrons per cubic cm., and m is the mass of one electron,

the energy of the electrons is practically independent of the temperature. The maximum momentum is determined by the total energy of the system, that is, momentum space is contained in a sphere of radius $\sqrt{2\pi E}$, where E is the total energy. But the number of cells in this space is determined by the volume of each which is $\frac{h^3}{V}$. The smaller the cells the more there are of them, that is, the greater the dilution V the more cells available in momentum space, and the less liklihood there is of two electrons being in the same cell. When they are in different cells, magnetic spins need not cancel, so that increased dilution tends to make the electron spins independent of each other. Therefore, at great dilutions, or at high temperatures, the Fermi statistics goes over to the Boltzman statistics, where the electrons are completely independent magnets.

Sommerfeld (4) applied the Fermi theory to metallic phenomena. He investigated the distribution curve and found, that at room temperatures and higher, the distribution function departs very little from the absolute zero temperature form. He was thus able to interpret such phenomena as specific heat, thermionic emission, photo-electric effect, etc., by imagining an absolute zero distribution.

Bloch (5) added to the theory by considering, first the motion of an electron in a potential field, and secondly, how the

electrons interact with the atomic lattice. Bloch was able to show that even if the positive ion centers are taken into account, the coupling and uncoupling of magnetic spins would not differ much from the distribution of the free electron gas. Thus the chance for finding in nature a test for the statistical theories is enormously increased.

In 1864 Weyl (6) observed that sodium and potassium were soluble in liquid ammonia. A very extensive study was made of the properties of these solutions by Kraus (7) and his co-workers.

A dilute solution of sodium in liquid ammonia has a characteristic blue color. It conducts electricity very much like a salt solution. The explanation given by Kraus (8) for the phenomenon is that there exists in such a solution an equilibrium between the unionized metallic atom, and its positive ion and the negative electron:

$$Na \longrightarrow Na^{+} + {}^{-} .$$

A concentrated solution has a bronze mettalic lustre and has the appearance of liquid metals. Indeed, a saturated solution of sodium in liquid ammonia has a greater electrical conductivity than solid iron. L. Farkas (9) has made a theoretical study of these conductivities.

APPARATUS AND METHOD

The best known method for measuring magnetic susceptibilities at low temperature employs the Gouy principle. The apparatus used was similar to that described by Freed and Kasper (10). A glass tube G (Figure 2) with a glass partition P is placed in a magnetic field (MM are the pole pieces of the magnet) so that P is in the center of the pole gap. The portion of the tube above P is filled with the solution of sodium in liquid ammonia, and that below P is filled with the pure solvent. The force which a magnetic field exerts on such a tube is $F = \frac{1}{2} A X (H^2 - Ho^2)$, where F is the force, A is the net cross-section of the substance, and X is the susceptibility per unit volume.

Fig. 2

H is the magnetic field at the pole gap, while Ho is the field at the tube extremities.

The above formula holds when there is a vacuum below the partition P. By placing the pure solvent in the lower end of the tube, a procedure used by Freed and Kasper (10), the effect of the magnetic field on the solvent of the solution is cancelled out, except for a small correction due to the solvent displaced by the solute. In this way only the force which the magnetic field exerts upon the dissolved solute is measured. The forces are measured by suspending the glass tube from the stirrup of a micro-

balance.

The diameter of the tube G had to be known and it was determined by measuring with a microscope the lengths of the tube which known weights of mercury would fill. Reducing the temperature of the tube has generally been accomplished by putting it in contact with condensed gases, such as liquid nitrogen, liquid propane, etc. Intermediate temperatures have been obtained by reducing the temperature under which the liquids boiled. Such a reduction in pressure necessitated enclosing the entire system in a vacuum-tight container. The method of Freed (11) avoids these complications.

The glass tube G containing the sodium solution was suspended in a brass tube A (Figure 3) (about 12 mm. in diameter and about 70 cm. long) from the stirrup of a micro-balance. The glass tube was surrounded by a gas of known temperature with which it came into equilibrium. The gas streamed slowly through the small copper tube B of about 1 mm. bore into the bottom of A (there were actually two such tubes), and then out into the atmosphere at the upper end of A. C is the Dewar flask which contained the liquified gases for cooling. It had a flattened construction at D which fitted in between the pole pieces of the magnet. A rubber tube R fitted tightly around the Dewar tube stopper F, and also around Dewar tube C. F was covered with a mixture of beeswax and rosin. T, T, T, are the junctions of copper-constantin thermo-couples, on whose cold junctions were fused little buttons of silver, which obtruded slightly into A. The buttons were thermally and electrically insulated from the copper tube A by means of little bakelite plugs. The temperature calibration for these thermo-couples was taken from a comparison of such couples with the melting points and boiling points of some standard substances.

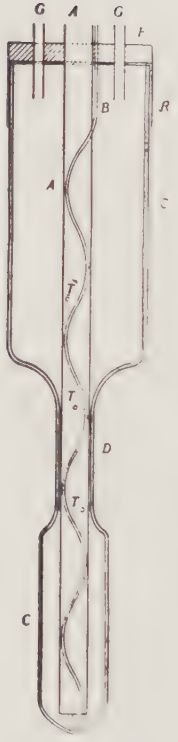

Fig. 3

Sufficient regularity in the flow of the gas stream was achieved by means of a capillary tube about 0.001 mm. in diameter. It delivered about 30 cc. of hydrogen gas per minute under normal conditions of pressure and temperature, when the pressure of the hydrogen tank was about one atmosphere above the external pressure. The upper ends of the little tubes B were made of monel metal to lessen the heat leak from the bath of condensed gas.

The cold dry gas having passed through A has sufficient time to become warmed through the copper and brass tube so that it leaves the apparatus above the dew point. Otherwise moisture would condense on the gold suspension and alter its weight. The suspension consisted of a light gold chain fastened to the glass tube by means of an aluminum tube (8 cm. long), which screwed on to an aluminum collar fixed on the glass. The purpose of the aluminum was to enable one to fill the tubes and seal them off above the collar and thus not alter the length of the suspension. In this way the tube would always hang in the same portion of the magnetic field.

The operation is carried out as follows. Liquid propane is introduced through G and C (Figure 3). A stream of dry hydrogen passing through B into A reaches the temperature of the liquid propane, and then comes into contact with the glass tube containing the sodium solution which is suspended in A. The gas finally passes out into the atmosphere through the top of A. To reduce the temperature of the sodium solution, a large vacuum pump is connected to G and the temperature is lowered since the liquid propane is now boiling under reduced pressure.

The solution tube comes into contact with the colder hydrogen and the magnetic susceptibility is measured at this temperature. The electro-magnet used for the work is described by Kay (12).

A maximum field of about 18,000 gauss was used.

Liquid Ammonia Apparatus and Manipulation

The apparatus employed in making up the sodium solutions is shown diagrammatically in Figure 4. The apparatus is first evacuated to about 10^{-5} mm. of mercury and then flamed to drive off adsorbed moisture. Pure sodium, found to have less than one part per million of iron, was introduced at the bottom of tube A through a capillary, a method described by Kraus (13). The ammonia supply was contained in a metal cylinder in which it was treated with metallic sodium. This cylinder was connected to the tube J. To make absolutely certain that the ammonia was dry, it was condensed into tube D which contained metallic sodium. Since it was necessary to determine the concentration of the solutions, the ammonia introduced into cell A was measured before introduction. To do this it was condensed into a measuring cell K, described by Kraus (14), which consisted of the chamber L and two graduated tubes MM. These tubes were about 10 cm. in length and had a total capacity of about 1 cc. The cell having been calibrated the volume of ammonia could be determined within one one-hundredth of 1 cc. The measuring cell was immersed in boiling ammonia during the process of condensation. When the desired amount of ammonia had been condensed into K, stopcocks H, L, C, were closed and B opened. The ammonia was then condensed over into tube A. Tube A contained a piece of soft iron sealed into pyrex glass. By means of a solenoid, S, the solution could be stirred by moving the solenoid up and down over tube A.

Approximately 15 cc. of ammonia were introduced into A. The tube was so designed that its volume up to the small capillary tube T, was about 5 cc. If a dilution was desired approximately 10 cc. of the solution the ammonia could be siphoned over into

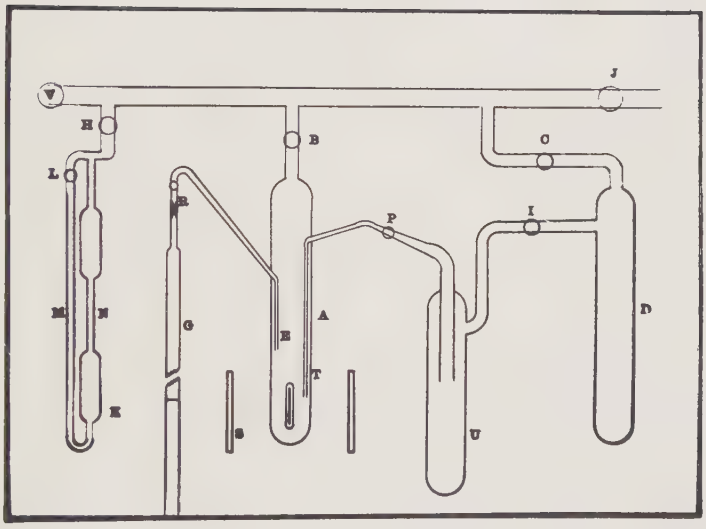

Fig. 4.

tube U through the small capillary tube T, by merely reducing the temperature of the tube U below the boiling point of liquid ammonia. The exact amount of liquid ammonia siphoned over was determined by condensing the ammonia from U to a 10 cc. measuring cell similar to K. This ammonia was then introduced into tube A again. Thus the concentration was reduced by two-thirds on each dilution. The sodium in U was determined by weighing it as sodium sulphate in platinum crucibles. When the solution obtained was of the concentration desired, enough solution was siphoned over through the capillary E to fill the tube G above the partition P. It was then sealed off at a construction R.

The section of the tube below P was then sealed to a vacuum line and filled with liquid ammonia. A small trap at the lower end of tube G prevented a bubble of gaseous ammonia (which is always present when the tube is sealed off the line), from rising to the partition P when the tube is in an upright position.

Before placing the tube G into the tube A of the magnetic field apparatus (Figure 3), it was necessary to remove all ice from the outside surface of the tube, otherwise it took hours for the tube to come to constant weight in the slow stream of dry hydrogen. To do this, the tube was washed in dry liquid ammonia and immediately placed into a glass apparatus surrounded by liquid ammonia. This apparatus was then evacuated for several hours. The tube was then transferred to the magnetic field apparatus in a stream of dry propane by means of a specially constructed dewar flask.

Method of Measurement

The glass tube G was magnetically calibrated at room temperature with distilled water. The lower part of the tube was evacuated while the top was filled with distilled water. The change in weight suffered by the tube was observed as definite

amperages were going through the coils of the magnet. The change in weight was plotted against the corresponding amperages. A similar curve was obtained for the same tube with both sides evacuated. This gave the zero correction for the corresponding amperages. Equal currents imply equal field strengths. Tube G was then filled with a sodium solution above and the pure solvent below. The zero correction in this case was determined by measuring the change in weight suffered by the same tube filled with liquid ammonia on both sides of the partition. In each case the change in weight of the tube was plotted against the amperage. By subtracting the ordinate of the zero curve from the ordinate of the solution curve the net change due to the magnetic field was obtained.

Comparisons were made at different temperatures. Some typical curves are plotted in Figure 5.

The method of calculation is as follows:

$$F_w = \tfrac{1}{2} A X_w (H^2 - Ho^2)$$

$$Fn_x = \tfrac{1}{2} A Xn_x (H^2 - Ho^2)$$

Ho^2 measured and found negligible.
Where F is the change in weight due to field H.
A is the cross-section of material.

X is the magnetic susceptibility per unit volume.

w refers to distilled water.

n_x refers to sodium and displaced ammonia.

Xn_x is the susceptibility per unit volume due to sodium present corrected for the amount of ammonia displaced.

$$\frac{F_w = \tfrac{1}{2} A X_w (H^2 - Ho^2)}{Fn_x = \tfrac{1}{2} A Xn_x (H^2 - Ho^2)}$$

$$\therefore \quad Xn_x = \frac{Fn_x}{F_w} \cdot X_w$$

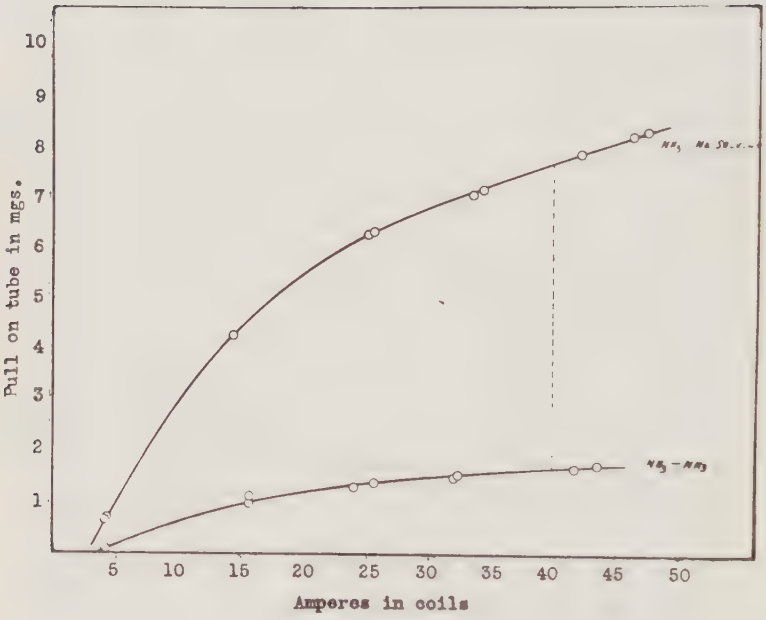
Pull on tube in mgs.
10
9
8
7
6
5
4
3
2
1
5
10
15
20
25
30
35
40
45
50
Amperes in coils

Fig. 5.

But

$$Xn_x = XNa \text{ (grams of Na)} + XNH_3 \text{ (grams of } NH_3 \text{ displaced)}$$

where XNa is the susceptibility per gram of sodium XNH_3 is the magnetic susceptibility of ammonia per gram

or solving for XNa

$$XNa = \frac{Xn_x - (XNH_3 \text{ x grams } NH_3 \text{ displaced})}{\text{grams of Na}}$$

The susceptibility per mol of sodium will be,

$$X/\text{mol Na} = XNa \text{ x atomic weight}$$

It was necessary to know the density of liquid ammonia at different temperatures as well as the density of the sodium solutions for different concentrations. These values were obtained from the literature (15, 16).

EXPLANATORY

The electrons in completed shells of the atom have neutralized each other's magnetic moment. Their contribution is a diamagnetic effect proportional to the area swept out by the electrons. Pauling (17) estimates the atomic susceptibility of Na^+ to be -4.2×10^{-6}. This value however does not come into the calculations of the experimental measurements.

The paramagnetic susceptibility is associated with a permanent magnetic moment of a substance, and is dependent on the temperature. Langevin gave the following simple relation

$$X_{Par} = \frac{N\mu M^2}{3KT}$$

where M is the magnetic moment of the atom

and $N = (6.06 \times 10^{23})$ Avogadros number

According to the quantum theory the angular momentum J is a constant of the motion of the atomic system, that is, for a fixed energy there is a fixed angular momentum. In the simple case the magnetic moment from the classical theory is given by

$$Y = \frac{eh}{4\pi mc} \quad J.$$

Where c is the velocity of light.

When $J = 1$ $\quad Y = \frac{eh}{4\pi mc}$, which is known as the Bohr magneton.

RESULTS

Coil amperage. 40
Temperature. . . . . 43°C
Pull exerted on tube with
distilled water. . . . . 0.022 grams
Area of cross-section. . . .2020 sq. cm.

TABLE I*

Tube	Concentration in Mols per Liter at -43°C	Density of Solutions at -33.8°C	Net Pull in Grams Exerted on Tube	Molal Susceptibility of Sodium	Per Cent Probable Error
Pure Na	--	--	--	(12-16) x 10^{-6}	
1	0.47	.6758	0.00063	9 x 10^{-6}	30%
2	0.1657	.6801	0.000602	84.4 x 10^{-6}	2
3	0.0174	.6818	0.000190	291. x 10^{-6}	5
4	0.010	.6820	0.000123	370. x 10^{-6}	8
5	0.0021	.6821	0.000073	1064 x 10^{-6}	15
6	0.0015	.6821	0.000055	1055 x 10^{-6}	15

*1 Bohr unit is equivalent to about 1600 x 10^{-6} units of magnetic susceptibility.

Changes in weight were determined to within $\pm$ .000005 grams.

Temperature Coefficient

40 amperes in coils

Solution same as tube 2, Table I

TABLE II

Temperature	Concentration in Mols per Liter	Pull in Grams Exerted on Tube	Density	Susceptibility per Mol of Sodium
248.2°A	0.1622	.000995	.6711	165.4
230.0°A	0.1657	.000602	.6936	84.4
220.0°A	0.168	.000380	.7056	39.88
209.2°A	0.171	.000263	.7182	17.0
203.8°A	0.173	.000230	.7245	10.83

In determining the density concentration in column two it was assumed that the density of the solution varied with the temperature in the same way as pure liquid ammonia. Although these results may be in error by 3 per cent, the relative accuracy for the different temperature is very much better. The temperatures are known to ± 0.2 degrees.

TABLE III

40 amperes in magnet coils

Tube	Temperature	Concentration in Mols per Liter	Density	Pull in Grams	Molal Susceptibility of Sodium
5	230.0°A	0.0021	0.6936	0.000073	1064×10^{-6}
5	206.8°A	0.0022	0.7160	0.000078	1075×10^{-6}

The increase in pull was determined with a precision of $\pm 1 \times 10^{-6}$ grams.

TABLE IV

Dilution	Number of Electrons per Cubic Cm.	Wc Critical Energy	$\frac{Wc}{K} = T$	
Pure Na	2.9	5.6×10^{-12}	40,410.0	°A
1N	6.06×10^{20}	4.2×10^{-13}	3,066.00	°A
.1N	6.06×10^{19}	8.38×10^{-14}	612.0	°A
.02N	1.2×10^{19}	3.09×10^{-14}	225.7	°A
.01N	6.06×10^{18}	1.95×10^{-14}	142.3	°A
.001N	6.06×10^{17}	4.2×10^{-15}	30.6	°A

Table IV shows a calculation from the Fermi theory. The critical energy was calculated for various values of the dilution parameter (the electron density). Equivalent temperature magnitudes were calculated in column four. At 0.1N (0.1 mols of sodium per liter), the critical temperature is about 600°. At 0.02N the corresponding temperature is about 225°C which happens to be within the temperature range of liquid ammonia.

The results show the atomic susceptibility of sodium in a .47N solution to be about the same as in metallic sodium. In a 0.0174N solution the atomic susceptibility of sodium is about 30 times that of metallic sodium. The curve in Figure 6 shows a rapid increase in the atomic susceptibility of sodium as we decrease the concentration. The curve is very steep near 0.01N. The electron density must be very near the critical point where the electrons uncouple and become independent magnets. The parallelism with the theory is very striking.

At 0.002N the atomic susceptibility is one hundred times that of pure sodium or almost two-thirds of a Bohr unit.

Table II gives the molal susceptibility of sodium in a 0.165N solution obtained for different temperatures. Variations in the molal susceptibility are plotted against temperature (Figure 7).

The magnetism of the sodium decreases with decreasing temperature, approaching the molal susceptibility of metallic sodium in the neighborhood of the liquid ammonia freezing point. This result is very interesting. It may be due to the formation of aggregates or clusters near the freezing point of the solvent, in anticipation of the metallic sodium crystallizing out.

In extremely dilute solutions, where the sodium atoms are far apart, this formation of clusters would be more difficult. The results obtained in Table III show that at .0021N the atomic susceptibility of sodium increases with lower temperatures, or that Curie's law is obeyed qualitatively. The increase in atomic susceptibility is of the right order of magnitude. This means that in very dilute solutions the electrons behave as independent magnets.

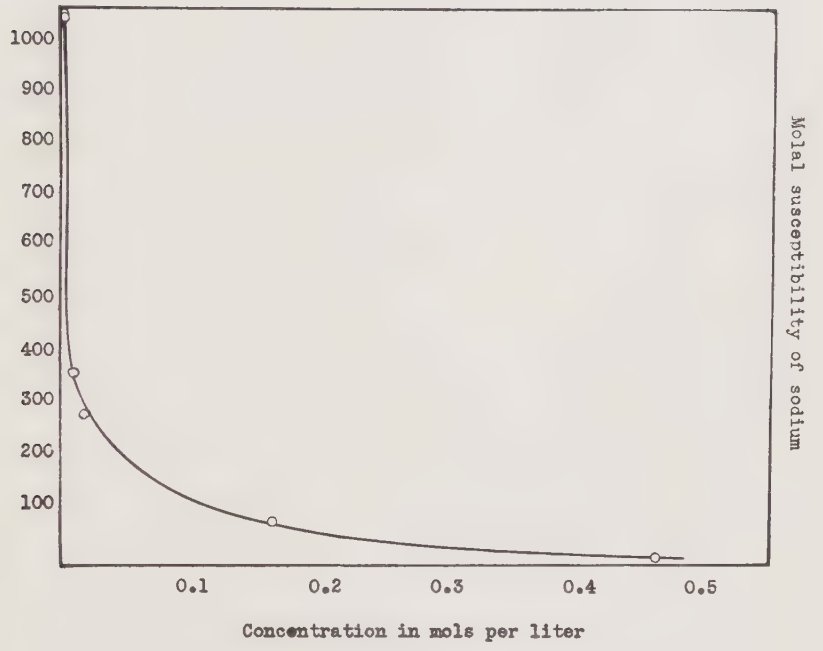
1000
900
800
700
600
500
400
300
200
100
0.1
0.2
0.3
0.4
0.5
Molal susceptibility of sodium
Concentration in mols per liter

Fig. 6.

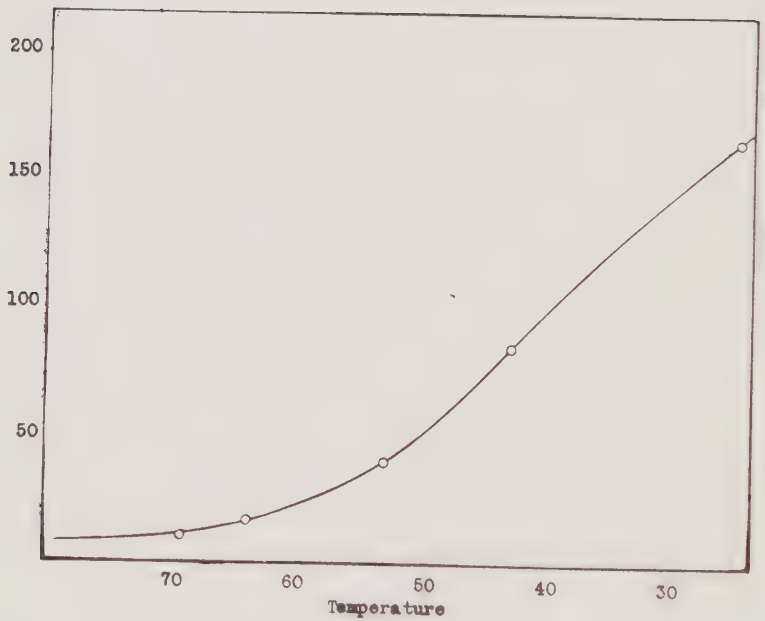

Fig. 7.

SUMMARY

It was a signal contribution to the chance of an experimental test of the Fermi-Dirac statistics when Bloch (18) included the interaction of the electrons with the lattice in the representation of the metal. This representation is much nearer to actual metals than an abstraction such as a free electron gas. Fortunately, the theory leads to a paramagnetism of the metal electrons which does not differ radically from the paramagnetism of free electrons, approximating the latter as the binding between the positive ions and electrons becomes weaker.

The distribution of spin and the magnetic susceptibility, as derived with the Fermi-Dirac statistics, are functions of the molal volume and the temperature in addition to the degree of binding at a given temperature an increase in the molal volume will lead the distribution from the degenerate state where practically all the electrons have paired off their spins to a state where the individual spins are independent of each other, the state where classical statistics are valid. The same general course is followed when the temperature is raised at a fixed volume.

In actual metals, the differences in molal volume are too slight to make much difference in the distribution of energy, or of spin among the electrons. Consequently, any change in distribution with temperature would become marked only above the boiling points of the metals.

This research consisted in diluting a metal with a non-metal, the non-metal having its electrons so tightly bound and

magnetically neutralized that in the first approximation the paramagnetism can be ascribed to the electrons of the metal. In this way a great variation in molal volume can be studied, and the temperature where considerable changes in the distribution of spin occur, is brought within range for experiment.

The magnetic susceptibilities of sodium dissolved in liquid ammonia were measured in this work although this precise situation was not treated by Bloch. However, it was hoped that the data would parallel the results of the theories of Pauli and Bloch. As a guide in the selection of substance, the well known fact that alkali and alkaline earth metals conduct electrolytically in dilute solutions and conduct metallically in concentrated solutions was depended upon. The concentrated solutions have every appearance of liquid metals. Indeed, a saturated solution of sodium, for example, has a greater electrical conductivity than solid iorn.

L. Farkas (19) has made a theoretical study of these conductivities. There is here, it seems, the gradation in metallic properties desired.

A rough computation on the bases of a free electron gas shows that metallic sodium could reach the critical temperature at about 40,000°A. That is, its electrons would begin to uncouple in marked degree and pass into a state where they act as independent elementary magnets. At 0.1N (0.L mol per liter), the critical temperature is about 600°A. At 0.02N the corresponding tmperature is about 225°A which happens to be within the temperature range of liquid ammonia. At .01N the temperature is about 140°A and at .001N about 30°A. Measurements made at 230°A show that at about 0.5N the atomic susceptibility of sodium has the same order of magnitude as pure metallic sodium. At .017N it has about thirty times the atomic susceptibility of pure sodium, and at .0022N it has about one hundred times the susceptibility of

pure sodium, now possessing about two-thirds the paramagnetic susceptibility which would arise from independent magnets having one-half unit of spin.

In a 0.165N solution the atomic susceptibility of sodium was found to decrease with lower temperatures, Near the freezing point of liquid ammonia the atomic susceptibility approaches that of sodium metal. This may be evidence for the formation of aggregates in anticipation of crystallization. At 0.002N the atomic susceptibility increased with lower temperatures. The order of magniture of this increase was that to be expected from Curie's law, which implies that the elementary magnets are finally almost independent of each other.

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